Scientists Perform First Neutron-Scattering Experiment Using the 11-T Superconducting Magnet at the Lujan Neutron Scattering Center

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During the 2002 run cycle at the Los Alamos Neutron Science Center, scientists successfully completed the first neutron-scattering experiment using the 11-T Oxford superconducting magnet on the Asterix spectrometer — an instrument designed for studies of magnetic materials. As a consequence of a special October 2002 call for proposals for experiments requiring high-magnetic fields, an experiment involving the order-parameter measurement of a three-dimensional, random-field Ising model, as realized in a dilute antiferromagnet, was selected. The intent of the experiment was to measure how the sublattice magnetization of the dilute antiferromagnet changes with temperature near the phase transformation. Magnetic ordering in this system results within the formulism of the Ising model when the magnetic moments align themselves in one of two anti-parallel directions.

Our experiment was noteworthy for several reasons. First, neutron beams in bulk single crystals have often confounded order-parameter measurements of these systems with neutron scattering. To overcome this problem, the researchers used a 1-cm- by 1-cm- by 250-nm-thick $Fe_xZn_{1-x}F_2$ (where $x > 0.754$) single crystal as their sample. Because the sample was so thin, extinction of the neutron beam was minimized. Secondly, a 10-T (or more) magnetic field was needed to cause a crossover to the critical behavior (i.e., the behavior of magnetic spin near a phase transition as predicted by theory and computer models and measured experimentally) observed in the random-field Ising model. Thirdly, precise and accurate control of the thermal environment to better than 5 mK was needed in the 63.8-K thermal transition region. With this experiment, we demonstrated the capability of Asterix to obtain high-quality data from exceedingly small samples at high fields under extremely stable thermal conditions.

Achieving Thermal Stability in an Unstable Temperature Region

Our most challenging technical problem was to obtain thermal stability in a temperature region where the Oxford cryostat is most unstable. Near 60 K, the measured temperature stability of the Oxford cryostat is on the order of 50 mK when better than 5 mK is needed. We solved this problem by connecting a spare calibrated carbon-glass thermometer (i.e., with the fiber axis parallel to the field axis of the magnet) at the sample location to a Lakeshore temperature controller. We then used the Lakeshore controller to regulate the sample temperature with a second heater wired to the sample stage (Fig. 1). With this arrangement we could change the temperature of the sample in a completely monotonic fashion. During subsequent neutron measurements, which typically lasted between 30 and 120 minutes, we measured the sample temperature to acquire a thermal distribution. The standard deviation of the distribution was typically less than 0.3 mK — much less than the 5-mK requirement.

During the two-week experimental run, we used Asterix to measure the intensity of the (100) Bragg reflection from the antiferromagnetic order in the $Fe_xZn_{1-x}F_2$ crystal sample. Because the d-spacing of the (100) Bragg reflection is large, we used ~ 6-Å neutrons to measure this reflection with the Asterix spectrometer in the unpolarized mode. This setup minimized background scattering from the aluminum in the magnet cryostat (Fig. 2), the variable temperature insert, and the sample holder. To further reduce background scattering, we inserted borated polyethylene neutron shields between the magnet and the two-dimensional, position-sensitive-detector (PSD) array (Fig. 2). The stepper-motor-controlled goniometer and sample stick allowed us to align the sample within about one minute after it was inserted in the magnet. (Prealignment of the sample with a Laue camera was not performed.)

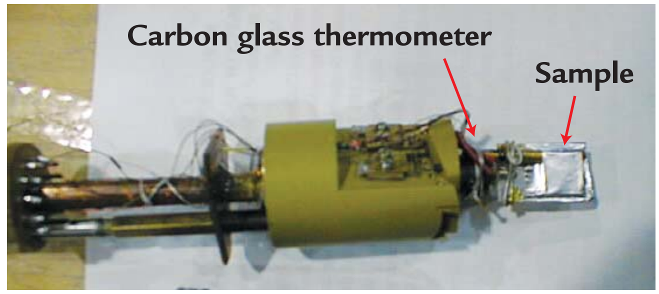

Fig. 1. View of the end of the sample stick for the 11-T Oxford superconducting magnet. The gold-colored object is a stepper-motor-controlled goniometer used to quickly align the sample. To achieve 0.3-mK thermal stability, we attached a carbon-glass thermometer and heater wire to the sample holder and used a Lakeshore temperature controller to control them.

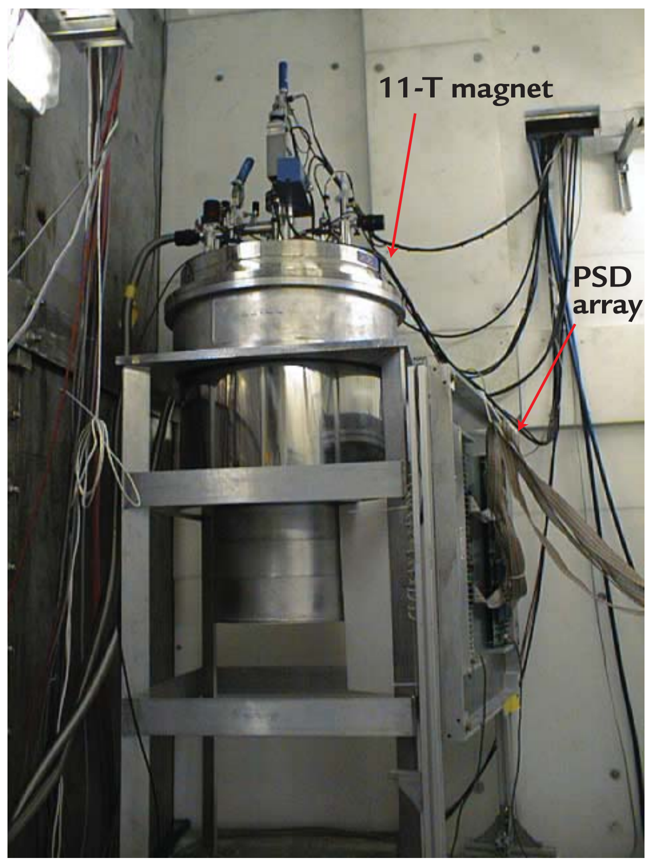

Fig. 2. View of the 11-T Oxford superconducting magnet during the neutron-scattering measurement. To the right of the magnet is the Asterix two-dimensional PSD array. Between the magnet and the detector, we installed borated polyethylene shields, which helped reduced the neutron background by one order of magnitude.

Fig. 3 shows an image of the intensity of neutron scattering as a function of scattering angle (i.e., in the equatorial plane of the magnet) and wavelength taken with a magnetic field of 10.000 ± 0.001 T and a sample temperature of 50.0000 ± 0.00003 K in 30 minutes (i.e., with a 120-µA target current). The arrow points to the (100) Bragg reflection that resulted from antiferromagnetic ordering in the sample. The mass of the sample material contributing to this reflection is about 100 µg (its volume is about 0.025 mm^3). The intensity of the reflection is plotted in Fig. 4 as a function of temperature for two of the six measurement conditions investigated. The first measurement is of the sample after it was cooled in a zero magnetic field and then warmed up through the transition (in zero field). The second measurement is of the sample after as it was warmed up in a 10-T magnetic field after first being cooled in a zero magnetic field.

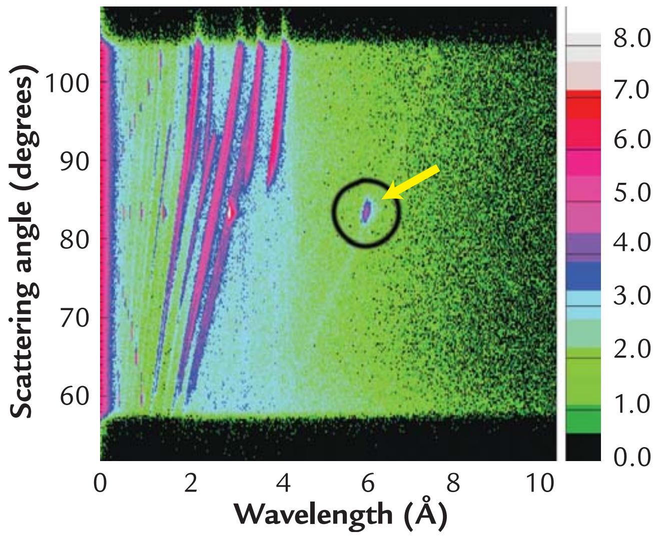

Fig. 3. An intensity image of neutron-scattering data plotted versus scattering angle and wavelength. The arrow points to the (100) Bragg reflection that resulted from antiferromagnetic ordering in the sample.

Conclusion

We have succeeded in obtaining data that provides insight into critical behavior predicted by the random-field Ising model. We have also demonstrated that Asterix can obtain wide-angle diffraction data from a single-crystal, thin-film sample in a timely manner; can obtain measurements from a sample in the presence of high magnetic fields; and can maintain an extremely stable thermal environment. Interestingly as well is the juxtaposition of the roles for magnetic scattering using x-rays and neutrons. Extinction of the neutron beam would confound order-parameter measurements for some bulk single crystals, whereas the problem was successfully studied with neutron scattering using a thin-film single crystal. Magnetic scattering of x-rays was successful at solving a similar problem using a bulk single crystal.[1] However, this technique failed in a thin-film-crystal study because the

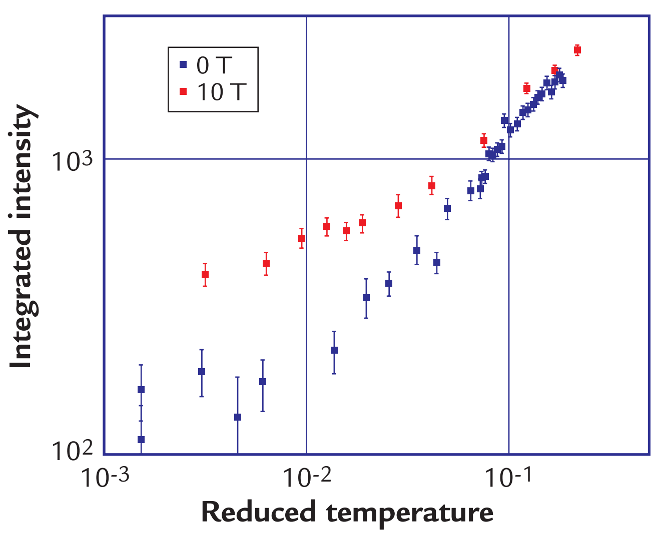

Fig. 4. The intensity of the (100) Bragg reflection from $Fe_xZn_{1-x}F_2$ after the sample was cooled in a zero magnetic field. The intensity was then measured as the sample was warmed up through the Néel transition in a zero magnetic field (●) and in a 10-T magnetic field (■). The latter condition is one that promotes detection of critical behavior in the random-field Ising model.

charge scattering from the forbidden Bragg reflection from the surface of the non-magnetically ordered MgF_2 single-crystal substrate (on which the $Fe_xZn_{1-x}F_2$ crystal is grown) is superimposed on and overwhelms the x-ray measurement of the (100) Bragg reflection from antiferromagnetic order in $Fe_xZn_{1-x}F_2$.

Acknowledgements

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References

1. F. Ye, L. Zhou, S. Larochelle, L. Lu, D.P. Belanger, M. Greven, and D. Lederman, "Order Parameter Criticality of the d = 3 Random-Field Ising Antiferromagnet $Fe_{0.85}Zn_{0.15}F_2$," *Physical Review Letters* **8915** (15), 7202 (2002).